



## Effect of morphology on water sorption in cellular solid foods. Part II: Sorption in cereal crackers

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### ARTICLE INFO

#### Article history:

Available online 1 September 2011

#### Keywords:

Network model  
Porous media  
Moisture sorption  
Cellular structure  
Effective diffusivity  
Cereals  
MRI

### ABSTRACT

Experimental dynamical moisture profiles of crackers with a fine and coarse morphology are successfully predicted using a pore scale network model. Experimental profiles are obtained using a single point imaging (SPI) NMR technique that enables 3D mapping of the moisture content of relatively immobile water at low water activity.

The relative vapor conductivity through the structure is 33% and 64% for the fine and coarse structured crackers, respectively. It can be argued that this is due to their difference in cell connectivity and not directly related to their difference in average cell diameter (0.33 and 0.75 mm, respectively). It was found that the retarded local sorption dynamics of the solid matrix has a noticeable influence on the moisture profiles that arise in the first hours. This is crucial for the moisture sorption dynamics of sub centimeter size samples, for which there is a distinct non-equilibrium between the vapor and the sorbed water phase. The local sorption at low water activity is a factor 3 faster for the fine structure cracker compared to the coarse one. This is due to their differences in average lamellae thickness (54 and 93 μm, respectively).

However, for the description of the overall moisture sorption dynamics of the few cm thick samples, on a time scale of days, it is valid to assume local equilibrium and to use an effective diffusivity model. The relative vapor conductivity together with the porosity and the derivative of the sorption isotherm determines the effective moisture diffusivity for these open structures, which is a factor 3 lower for the fine structured cracker compared to the coarse one. The single sided moisture sorption in the 2.5 mm thick cracker samples is not even completed after 5 days, mainly because at higher water content (near 20%) there is very little gradient in relative humidity to drive the vapor transport. This is reflected in the predicted effective moisture diffusivities which for the coarse cracker decrease from  $16 \times 10^{-9} \text{ m}^2/\text{s}^2$  (at 1% MC, 16%  $a_w$ ) to  $7.6 \times 10^{-10} \text{ m}^2/\text{s}^2$  (at 20% MC, 86%  $a_w$ ).

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### 1. Introduction

The ability to predict dynamic moisture profiles of porous cellular foods during drying, wetting, or storage is of great importance for the food industry. For example, crispness is one of the most important quality attributes of foods like cereals, bread, crackers, and snacks. It is quickly lost when the water activity exceeds about 0.5. For bread, consisting of a dry crispy crust and a moist soft crumb, crispness of the crust is lost within a few hours after baking due to water migration from the crumb to the crust. More insights in the

controlling factors of water migration would enable the industry to manufacture products with a prolonged crispness.

The aim of this study is to gain more insight between the structural characteristics and the moisture profiles which arise in a sample when it is exposed to an environment with a higher relative humidity. We developed a network model to predict the water sorption behavior of porous cellular solid systems. The moisture migration in cereal cellular solid systems involves diffusive transport in the gas phase and in solid phase. Both depend on the morphological details of the sponge structure, but in a different way. The model is based on the actual pore structure captured with X-ray tomography (XRT) and the sorption kinetics of the solid matrix deduced from dynamic Environmental Scanning Electron Microscopy (ESEM) experiments. The theoretical background, image analysis and mathematical

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implementation of the network model is described in Part I of this paper (Esveld et al., 2012). In this paper (Part II), we present the simulation and measurement of the dynamics moisture profiles in crackers with different morphology. The non-destructive monitoring during a sorption experiment of days was possible due to advances in single point imaging nuclear magnetic resonance (SPI-NMR), which captures the spatial distribution of the sorbed water even at low water activities. This allows validation of the network model and the assumption therein.

In order to generalize the results and to make them applicable in food product design, it is required to extract the characteristics embedded in the complex network model by reduction to a conventional volume averaged description. This connects with the practical approach in food science, where it is custom to lump the underlying transport phenomena in the porous medium to a single effective moisture diffusivity ( $D_{\text{weff}}$ ) as function of moisture content ( $w$ ) (Zogzas and Maroulis, 1996). Hereby the validity of Fick's second law (Crank, 1975) is assumed:

$$\frac{dw}{dt} = \nabla \cdot (D_{\text{weff}} \nabla w). \quad (1)$$

Unfortunately, they are difficult to estimate for a given composition and largely unknown structure (Vagenas and Karathanos, 1991). An excellent range of experimental studies on the rate of moisture sorption in porous cereal products has been published in recent years by Guillard et al. (2003a,b, 2004) and Roca et al. (2007, 2006). They have determined the effective moisture diffusivity from the moisture sorption kinetics in response to a small step increase of the surrounding relative humidity. Hereby it was assumed that the driving gradient for diffusion is the macroscopic moisture gradient in the typical 2 mm thick porous samples. This in contrast to a similar method for the determination of the moisture diffusivity in pharmaceutical powders (Burnett et al., 2006), where the gradient over the much smaller particle radii is used. In the latter method it is assumed that the macroscopic diffusion through the gas phase in the powder sample is sufficiently fast to exclude any macroscopic moisture gradient over the powder sample. Now both assumptions might be valid for the particular sample size, structure, and composition under study, but it raises the question how far these assumption can be stretched for other sample sizes and structures. The volume average parameters obtained from the volume averaged network model can be used for a separation of times scales of the macroscopic diffusion and local microscopic penetration in the solid matter. In this ways conclusions can be drawn about the sample structure and the size dependence, which are normally obscured in effective diffusivity fits (Tütüncü and Labuza, 1996).

### 1.1. From network model to effective water diffusivity

The network model contains the local structural details of the sample. This is especially useful to model the water sorption in cases of strong heterogeneity, such as when a crack or a crust is present. However in this study we have actually taken care to create homogeneous cracker samples. Thus it makes sense to extract the effective parameters for the averaged sample volume that rule the vapor transport and local sorption rate (see Part I (Esveld et al., 2012)). This allows to reduce the network model to a nearly equivalent continuous two equation model, which is reviewed below:

$$\varphi \rho_{\text{vsat}} \frac{d\bar{a}_v}{dt} + (1 - \varphi) \rho_{\text{sdry}} \frac{dw}{da} \frac{d\bar{a}_w}{dt} = \nabla \cdot (\gamma D_v \rho_{\text{vsat}} \nabla \bar{a}_v), \quad (2)$$

$$\frac{d\bar{a}_w}{dt} = \frac{\bar{a}_v - \bar{a}_w}{\tau}. \quad (3)$$

The balance Eq. (2) is written in a form which uses the activities of vapor and absorbed water as state parameters. It states that the accumulation of water in the air and solid phase is proportional to

the divergence of the vapor fluxes, which are driven gradient in vapor activity and proportional to the vapor in air diffusivity times the relative vapor conductivity  $\gamma$ . The water sorption from the vapor into the lamellae takes some time due to internal diffusion and visco-elastic relaxation. This was found from the swelling kinetics of small cracker particles to be typical exponential in nature. Therefore we can represent this mathematically by a first order relaxation of the absorbed water activity towards the vapor activity with a volume averaged typical time  $\tau$ , as stated in the second differential Eq. (3). Eqs. (2) and (3) must be solved simultaneously with for instance a finite volume discretization over space to get a volume averaged solution in time and space of the two state parameters  $a_v$  and  $a_w$ .

Now if we ignore the local sorption time and assume that  $a_v$  and  $a_w$  are in local equilibrium, that is  $\tau$  is insignificantly small and  $a_v$  and  $a_w$  are practically the same, than we can reduce Eqs. (2) and (3) to a single diffusion equation of the form (1). It then follows for the effective diffusivity (4) that it is notably ruled by the porosity  $\varphi$ , the relative vapor conductivity  $\gamma$  and the derivative of the sorption isotherm. It is by definition not influenced by the local sorption rate and thus also not by the water diffusivity in the solid matrix.

$$D_{\text{weff}} = \frac{\gamma D_v \rho_{\text{vsat}}}{(1 - \varphi) \rho_{\text{sdry}} \frac{dw}{da}} \quad (4)$$

## 2. Material and methods

### 2.1. Crackers

The crackers were made of 64% wheat flour (Zwaluw, 10.3% protein, Meneba, The Netherlands), 25% water, 0.12% salt, 0.12% yeast, 6.4% sunflower oil, 0.12% dextrose monohydrate, and 0.003% Bakers yeast B500. After mixing, the dough was rested for 20 min, laminated into slaps of 4.5 mm thick and proofed at 34 °C and 90% RH. Two different proofing times of 10 and 115 min are used to obtain two different kinds of crackers with different morphology. These samples are indicated as pt10 and pt115, respectively. The slabs were baked for 20 min at 170 °C in an air convection oven.

The structural details of the two samples are analyzed in Part I of this paper (Esveld et al., 2012). Their difference in fine structural scale can be appreciated from XRT data rendering in Fig. 1.

### 2.2. Sorption isotherm

Water sorption measurements of the crumbled crackers were performed at 25 °C using a symmetrical gravimetric analyzer (9 VTI Corporation SGA-100) equipped with an electronic microbalance and dew point analyzer. The samples were first equilibrated at the lowest RH and then the relative humidity was increased in 12 steps from 0% to 95%. The maximum equilibrium time was set to 3 h. The transient weight increase during each step was fitted with an exponential function to estimate the equilibrium value at infinite equilibration time as shown in Fig. 2. An overall fitted Oswin equation is used in the model, see Part I (Esveld et al., 2012).

### 2.3. SPI-MRI moisture mapping during sorption

The experimental moisture profiles in time were measured with a special NMR imaging (MRI) technique. Conventional MRI relies on the spin echo technique to measure the liquid water distribution (Hills, 1998; McCarthy et al., 1994). Spin echo is however not very suited for foods at low to intermediate water activity due to the short proton spin-spin relaxation times ( $T_2$ ) of the relative immobile water. With a single point imaging (SPI) technique it is possible to capture short  $T_2$  components within the

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